PATENT SPECIFICATION

Inventors: CHARLES OCKRENT and CHARLES SIMONS.

690,816

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COMPLETE SPECIFICATION

Improvements in or relating to the Manufacture of Substituted Phenoxy Acetic Acids

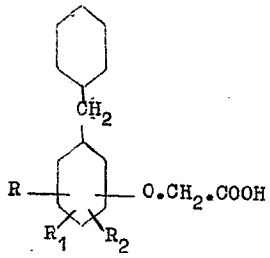
We, THE BRITISH DRUG HOUSES LIMITED, a British Company of 16—34, Graham Street, City Road, London, N.1, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention is for improvements in or relating to the manufacture of organic compounds and has particular reference to the manufacture of substituted benzyl phenoxy acetic acids.

Certain phenoxy acetic acids are known to act as plant hormones and are used *inter alia* as selective weed killers.

It is an object of the present invention to provide a process for the manufacture of a range of substituted benzyl phenoxy acetic acids.

According to the present invention there is provided a process for the manufacture of a substituted benzyl phenoxy acetic acid of the general formula:—



wherein R represents a chlorine atom or a methyl or ethyl group and R_1 and R_2 represent a hydrogen or chlorine atom or a methyl or ethyl group which process comprises reacting in a solvent the alkali metal salt of the appropriate phenol and the alkali metal salt of chloro-acetic acid, or an ester of chloro-acetic acid and when an ester is employed hydrolyzing the resulting esterified acid. The preferred alkali salts are the sodium salts.

The solvent employed may be any which is inert to the reactants and to the product of the reaction.

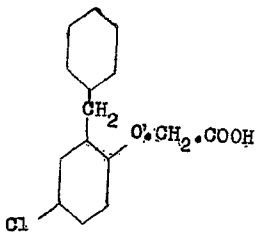
Thus, the solvent in which the alkali metal salt of the phenol reacts with the alkali metal salt of chloro-acetic acid may be for example water or alcohol and the solvent in which the sodium salt of the phenol and the ester of chloro-acetic acid are reacted may, for example, consist of toluene or dry ethanol.

The phenol may be prepared either by a method well known in the literature or it may be obtained by the method described in copending Applications Nos. 16329/49 (Serial No. 669,072) or 16340/49 (Serial No. 667,403).

Following is a description by way of example of methods of carrying the invention into effect.

EXAMPLE I.

2-Benzyl-*p*-chlorophenoxy acetic acid



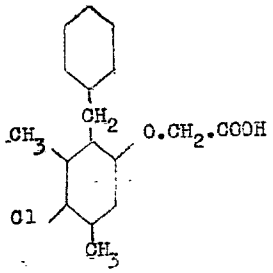
was produced by adding chloro-acetic acid (9.4 gms.) in 75% aqueous alcohol (100 ml.) dropwise to a refluxing solution of *p*-chloro-*o*-benzyl phenol (22.94 gms.) and potassium hydroxide pellets (6.5 gms.) in

75% aqueous alcohol (50 ml.). The mixture was refluxed for 12 hours and the alcohol removed *in vacuo*. The residue was diluted with water and acidified in Congo Red with mineral acid. The oil was extracted with ether and this ether extracted with sodium bicarbonate solution thus freeing the product from phenol.

Acidification gave a solid which is recrystallised from petroleum ether. M.Pt. 109° C.

EXAMPLE II.

2-Benzyl - 4 - chloro - 3:5 - dimethyl phenoxy acetic acid

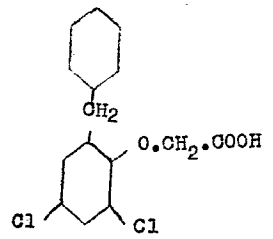


was prepared by dissolving sodium (2.3 gms.) in anhydrous alcohol (150 ml.) and 2-benzyl-4-chloro-3-5-xylen-1-ol (25 gms.) added. The mixture was refluxed and a solution of ethyl chloro-acetate (10.8 gms.) in ethanol (50 ml.) dropped in. After 10 hours refluxing caustic potash (14 gms.) was cautiously added and the mixture again refluxed for 1 hour.

The product (M.Pt. 153° C.) was isolated as described in Example I.

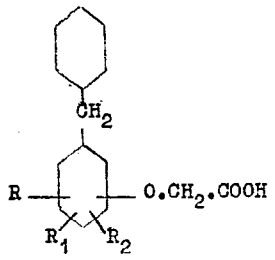
EXAMPLE III.

Following the general method of Examples I and II 2-benzyl-4:6-dichlorophenoxy acetic acid (m.p. 124° C.) was produced.



What we claim is:—

1. A process for the manufacture of a substituted benzyl phenoxy acetic acid of the general formula



wherein R represent a chorine atom or a methyl or ethyl group and R_1 and R_2 represent a hydrogen or chlorine atom or a methyl or ethyl group which process comprises reacting in a solvent the alkali metal salt of the appropriate phenol and the alkali metal salt of chloro-acetic acid or an ester of chloro-acetic acid, and when an ester is employed hydrolyzing the resulting esterified acid.

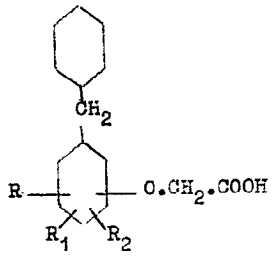
2. A process as claimed in claim 1 wherein the alkali metal salts are the sodium salts.

3. A process as claimed in claim 1 wherein the solvent for the alkali metal salt of the appropriate phenol and the alkali metal salt of chloro-acetic acid is water or alcohol.

4. A process as claimed in claim 1 wherein the solvent for the alkali metal salt of the appropriate phenol and the ester of chloro-acetic acid to toluene or dry ethanol.

5. A process for the manufacture of substituted benzyl phenoxy acetic acid substantially as described with reference to any one of the specific examples hereinbefore set forth.

6. A substituted benzyl phenoxy acetic acid of the general formula



wherein R represents a chlorine atom or a methyl or ethyl group and R_1 and R_2 represent a hydrogen or chlorine atom or a methyl or ethyl group.

BOULT, WADE & TENNANT,
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Chartered Patent Agents.

PROVISIONAL SPECIFICATION

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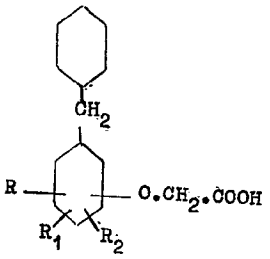

wherein the ring which contains the oxy acetic group may also contain as substituents R, R_1 and R_2 or any one or more of them, R, R_1 and R_2 each representing chlorine or ethyl or methyl which process comprises reacting in solution the alkali salt, e.g., the sodium salt, of the appropriate phenol and the alkali salt of chloro-acetic acid, e.g., the sodium salt of choro-acetic acid, or reacting in a solvent the alkali salt of the appropriate phenol with an ester of chloro-acetic acid.

The solvent employed may be any solvent which is inert to the reactants and to the product of the reaction.

Thus, the solvent in which the alkali salt of the phenol reacts with the alkali salt of chloro-acetic acid may be for example water or alcohol and the solvent in which the sodium salt of the phenol and the ester of chloro-acetic acid are reacted may, for example, consist of toluene or dry ethanol.

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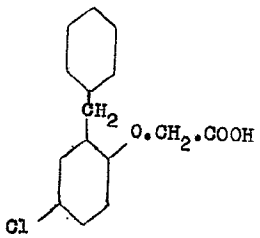

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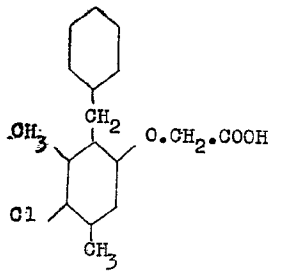

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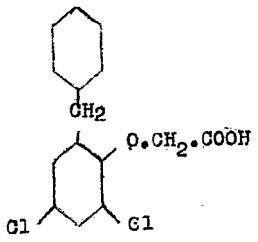

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